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FORM PTO 1390 (REV 11-2000)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER CHEMMT-206
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371			U.S. APPLICATION NO. (If known, see 37 CFR 1.5) <b>10/0888840</b>
INTERNATIONAL APPLICATION NO. <b>PCT/EP00/09193</b>	INTERNATIONAL FILING DATE <b>20 September 2000</b>	PRIORITY DATE CLAIMED <b>30 September 1999</b>	
<b>METHOD FOR APPLYING MANGANESE PHOSPHATE LAYERS</b>			
APPLICANT(S) FOR DO/EO/US <b>Klaus-Dieter NITTEL, Detlev SEIFERT and Ralf STICKLER</b>			
<p>1. <input checked="" type="checkbox"/> This is a <b>FIRST</b> submission of items concerning a filing under 35 U.S.C. 371.</p> <p>2. <input type="checkbox"/> This is a <b>SECOND</b> or <b>SUBSEQUENT</b> submission of items concerning a filing 35 U.S.C. 371</p> <p>3. <input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371 (f)). The submission must include items (5), (6), (9) and (21) indicated below.</p> <p>4. <input type="checkbox"/> The US has been elected by the expiration of 19 months from the priority date (PCT Article 31).</p> <p>5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371 (c)(2))</p> <p>a. <input checked="" type="checkbox"/> is attached hereto (required only if not communicated by the International Bureau).</p> <p>b. <input checked="" type="checkbox"/> has been communicated by the International Bureau.</p> <p>c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).</p> <p>6. <input type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371 (c)(2)).</p> <p>a. <input type="checkbox"/> is attached hereto.</p> <p>b. <input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4).</p> <p>7. <input type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))</p> <p>a. <input type="checkbox"/> are attached hereto (required only if not communicated by the International Bureau).</p> <p>b. <input type="checkbox"/> have been communicated by the International Bureau.</p> <p>c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.</p> <p>d. <input type="checkbox"/> have not been made and will not be made.</p> <p>8. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).</p> <p>9. <input type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).</p> <p>10. <input type="checkbox"/> An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).</p> <p><b>Items 11 to 20 below concern document(s) or information included:</b></p> <p>11. <input checked="" type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98.</p> <p>12. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.</p> <p>13. <input type="checkbox"/> A <b>FIRST</b> preliminary amendment.</p> <p>14. <input type="checkbox"/> A <b>SECOND</b> or <b>SUBSEQUENT</b> preliminary amendment.</p> <p>15. <input type="checkbox"/> A substitute specification.</p> <p>16. <input type="checkbox"/> A change of power of attorney and/or address letter.</p> <p>17. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.</p> <p>18. <input type="checkbox"/> A second copy of the published international application under 35 U.S.C. 154(d)(4)</p> <p>19. <input type="checkbox"/> A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).</p> <p>20. <input type="checkbox"/> Other items or information:</p>			

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I hereby certify that this correspondence is being deposited with the U.S. Postal Service by First Class Mail in an envelope addressed to: Commissioner for Patents, Washington, DC 20231, on the date shown below.

Dated June 25, 2002 Signature

*Eileen Sheffield*  
(Eileen Sheffield)

Docket No.: NY-CHEMMT-206-US  
(PATENT)

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

In re Patent Application of:  
Klaus-Dieter NITTEL, et al.

Application No.: 10/088,840

Group Art Unit:

Filed: March 21, 2002

Examiner: Not Yet Assigned

For: METHOD FOR APPLYING MANGANESE  
PHOSPHATE LAYERS

**PRELIMINARY AMENDMENT**

Commissioner for Patents  
Washington, DC 20231  
Box PCT

Dear Sir:

In advance of prosecution, please amend the above-identified patent application as follows:

**IN THE CLAIMS**

Please cancel claims 1-7, without prejudice and add the following new claims:

8. Method for applying manganese phosphate layers to iron or steel surfaces comprising contacting workpieces with a phosphating solution comprising
  - 0.2 to 4 g/l of iron (II) ions
  - 10 to 25 g/l of manganese ions
  - 25 to 50 g/l of phosphate ions (calc. As  $P_2O_5$ )
  - 3 to 35 g/l of nitrate ions
  - 0.5 to 5 g/l of nitroguanidine

said solution having 7 to 24 points of free acid, 50 to 140 points of total acid, and an S value of 0.2 to 1, wherein, after drying, the manganese phosphate layer having a minimum thickness of 2  $\mu\text{m}$  and an average maximum roughness depth ( $R^2$ ) of 2.5  $\mu\text{m}$

9. The method according to claim 8, wherein said phosphating solution that comprises 0.5 to 2 g/l of nitroguanidine.
10. A method according to claim 8, wherein the phosphating solution comprises not more than 2.5 g/l of iron (II) ions.
11. A method according to claim 8, wherein the workpiece is steel and said phosphating solution comprises a complex-forming agent for the alloying constituents of the steel.
12. A method according to claim 11, wherein said coupler-forming agent is citric acid.
13. A method according to claim 8, wherein said phosphating solution further comprises at least one of
  - 0.2 to 4 g/l of nickel ions and
  - 0.2 to 4 g/l of magnesium ions.
14. A method according to claim 8, wherein at least a portion of the manganese ions in said phosphating solution are replaced by manganese carbonate to neutralize free acid.
15. A the method according to claim 8, wherein said workpieces are subjected to a sliding friction.
16. A method according to claim 8, wherein said workpieces are selected from the group consisting of axles, gear mechanism parts and engine pistons.

**REMARKS**

The purpose of this amendment is to conform the claims to U.S. practice. Entry of this amendment is respectfully requested.

Dated: June 25, 2002

Respectfully submitted,

By

  
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### Method for Applying Manganese Phosphate Layers

- The invention relates to a method for applying manganese phosphate layers to iron or steel surfaces using
- 5 phosphating solutions containing manganese, phosphate, iron(II) ions as well as nitroguanidine, as well as its application to workpieces that are subjected to sliding friction.
- 10 On account of their high mechanical resistance, manganese phosphate layers have proved ideal for various applications, e.g. in order to reduce the friction of metal surfaces sliding on one another or to facilitate the cold forming of metals. However, relatively thick, coarsely
- 15 crystalline layers were obtained with the manganese phosphate solutions that were initially commonly used, these layers being particularly disadvantageous if fine mechanical parts are to be treated. Numerous proposals have therefore been made with the object of producing thin,
- 20 finely crystalline manganese phosphate layers. For example, it is known that an improvement in the phosphate layer can be obtained by adding condensed phosphates. Phosphating solutions based on manganese phosphate are however generally employed at high temperatures, which
- 25 means that, as a result of the considerable hydrolysis that takes place at high temperatures, the effectiveness of the condensed phosphates rapidly falls and/or replenishment condensed phosphate has to be constantly added.
- 30 Another way of obtaining fine-grain phosphate layers is described in German Auslegeschrift 1109 484. Nitrate-containing phosphate solutions in which the amount of nitrate is in excess of the amount of phosphate are used for this purpose. The solutions should have a ratio of
- 35 nitrate to phosphate of about 1.5-4.5 : 1. It has been

found however that in many cases the intended effect is not achieved.

Furthermore, a method is known in which an increased amount  
 5 of free acid in the phosphating solution is specifically  
 employed in order to obtain particularly thin layers (DE-C-  
 1246356). However, on account of their low surface density  
 these layers can, in practice, only apply to special cases.

10 Finally it is known to add to a phosphating solution based  
 on manganese phosphate or manganese-iron phosphate in which  
 the concentrations with respect to manganese, iron(II),  
 phosphate and nitrate ions lie within specific limits,  
 proportionally more free  $P_2O_5$  in relation to the total  $P_2O_5$   
 15 than corresponds to the phosphating equilibrium in the  
 working phosphating solution. The aforementioned measure  
 is said to have the advantages of achieving a significant  
 decrease in the amount of slurry formed in the phosphating  
 and a reduction of the chemicals needed to produce a  
 20 specific amount of coating (DE-B-22 13781).

A common feature of the known methods is that manganese  
 phosphate layers are formed having significant roughness  
 depths. The reason for this is that the etching action in  
 25 manganese phosphate systems is already pronounced at the  
 start of the process and leads to a punctiform removal of  
 metal after an extremely short action time. On the other  
 hand, the layer formation takes place relatively slowly  
 compared with zinc phosphate systems. The pronounced  
 30 etching action and delayed layer formation can be observed  
 visually by a large evolution of gas over a relatively long  
 period, the so-called gas time.

The object of the invention is to provide a method that  
 35 leads to manganese phosphate layers having as low a

roughness depth as possible, but whose layer thickness is in the medium to high range.

This object is achieved by the method of the type mentioned in the introduction and corresponding to the invention in which, in order to produce a manganese phosphate layer having a minimum thickness of  $2.5 \mu\text{m}$  and an averaged maximum roughness depth ( $R_z$ ) of  $2.5 \mu\text{m}$ , measured after drying, the workpieces are brought into contact with a phosphating solution containing

0.2 to 4 g/l of iron(II) ions  
10 to 25 g/l of manganese ions  
25 to 50 g/l of phosphate ions (calc. as  $\text{P}_2\text{O}_5$ )  
15 3 to 35 g/l of nitrate ions  
0.5 to 5 g/l of nitroguanidine

that has 7 to 24 points of free acid, 50 to 140 points of total acid, as well as an S value of 0.2 to 1.

The averaged roughness depth is defined according to DIN 4768, Sheet 1, and represents the arithmetic mean of the individual roughness depths of five mutually adjoining individual measurement stretches of identical length, defined as

$$R_z = 0.2 (Z_1 + Z_2 + Z_3 + Z_4 + Z_5)$$

The required maximum value of  $2.5 \mu\text{m}$  refers only to the roughness depth of the manganese phosphate layer and disregards the depth of the untreated metal surface.

The aforementioned total point number is determined in a manner known per se by titrating 10 ml of the phosphating solution after dilution with water to about 50 ml using



phenolphthalein as indicator, until the colour changes from colourless to red. The amount of 0.1 N sodium hydroxide solution used represents the total point number. Other suitable indicators for the titration are thymolphthalein  
5 and ortho-cresolphthalein.

The free acid points are determined in a similar way, dimethyl yellow being used as indicator and the titration being carried out until the colour changes from pink to  
10 yellow. Interfering metal ions are removed beforehand by adding hexacyanoferrate (II) or hexacyanocobaltate (III) ions. The S value is defined as the ratio of free  $P_2O_5$  to total  $P_2O_5$ . (For further details see W. Rausch, "Die Phosphatierung von Metallen", Eugen G. Leuze Verlag,  
15 Stuttgart 1974, pp. 273 ff.).

More particularly, it is known from GB-A-510684 to produce manganese phosphate layers using phosphating solutions that may also contain nitroguanidine in addition to numerous  
20 other oxidising agents. However, it can be calculated from the data relating to the point numbers of free acid and total acid that the phosphating solutions contain considerably lower concentrations of phosphating-active components and - corresponding to the objective pursued in  
25 the known method of improving the corrosion resistance of metals - can form layers of a very low layer weight. The patent specification does not contain any kind of information on the roughness depth of the phosphate layer.

30 The investigations carried out on the development of the present invention have shown that when using nitrate as accelerator, which autocatalytically forms nitrite as a result of the normally used high phosphating temperatures, or when using nitrite or chlorate, the layer formation is  
35 disturbed because of the deficient iron(II) content, or

layers are formed having only a very low layer weight or very low layer thickness. In contrast the use of nitroguanidine allows the iron(II) concentration to be kept below specific limits without resulting in an undesirable sharp drop in the iron(II) content necessary for the formation of a qualitatively high-grade layer.

In order to assist the oxidation of iron(II), oxygen-containing gas, for example compressed air, may be blown into the phosphating solution. Substances that oxidise iron(II), preferably potassium permanganate, may also be added. It should however be borne in mind that the iron(II) concentration should in no case fall below 0.2 g/l, since otherwise the desired layer weight will not be obtained.

A preferred embodiment of the invention envisages bringing the workpieces into contact with a phosphating solution that contains 0.5 to 2 g/l of nitroguanidine. Reasons of cost in particular are decisive in this connection.

Furthermore it is advantageous to adjust the concentration of iron(II) ions in the phosphating solution to a maximum concentration of 2.5 g/l. In this way, finely crystalline layers of small roughness depth can also reliably be formed in the case of workpieces that are difficult to phosphate.

If workpieces with steel surfaces are to be phosphated, a further advantageous embodiment of the invention envisages adding complex-forming agents to the phosphating solution in order to complex the alloying constituents of the steel. In particular chromium is such an alloying constituent. Suitable complex-forming agents are for example tartaric acid, but, in particular, citric acid. The constituents of

the steel that might adversely affect the layer quality are trapped by the addition of complex-forming agents.

A further advantageous modification of the invention  
5 consists in bringing the workpieces into contact with a phosphating solution that additionally contains

0.2 to 4 g/l of nickel ions

or

10 0.2 to 4 g/l of magnesium ions.

These additions produce an homogenisation of the etching attack on the metal surface to be treated and thereby achieve a stronger adhesion of the phosphate layer. Also  
15 the appearance of the phosphate layer is improved as a result of the generally desirable dark coloration. In addition, the content of magnesium ions reduces the overall consumption of chemicals.

20 Finally, it is expedient to contact the workpieces with a phosphating solution in which at least a proportion of the manganese ions have been replaced by manganese carbonate in order to neutralise the free acid.

25 The contact of the workpieces with the phosphating solution preferably takes place at a temperature in the range from 75° to 95°C.

The workpieces may be brought into contact with the  
30 phosphating solution in any appropriate way, preferably by immersion treatment. Treatment times of, in general, 1 to 15 minutes are appropriate.

As a rule it is necessary to clean the workpieces before  
35 the phosphating. Acidic, neutral or alkaline cleaning

agents are used for this purpose. In general the workpieces are thoroughly rinsed with water between the cleaning and the phosphating of the workpieces.

Particularly after treatment with alkalis and acids, the  
5 workpieces should be pre-rinsed in an aqueous slurry of finely divided manganese phosphate in order to promote the formation of particularly uniform finely crystalline layers in the subsequent phosphating.

10 Phosphate layers having a layer weight of in general 5 to 30 g/m<sup>2</sup> can be obtained by means of the method according to the invention.

The phosphate layers produced by the invention may, in a  
15 manner known as *per se*, be lacquered or provided with plastics coatings. In conjunction with corrosion prevention oils, these measures serve to increase the resistance to rust. The main application of the method according to the invention however is in the treatment of workpieces that  
20 are exposed to sliding friction. Such workpieces include, for example, axles, gear mechanism parts and pistons of internal combustion engines and compressors.

It is possible by means of the method according to the  
25 invention, to produce manganese phosphate layers with average to high layer thicknesses that, nevertheless, have only a very low averaged roughness depth. The roughness depth is ca. 30 to 50% below the values that were hitherto normally obtained. As a result of the low roughness depth  
30 the frictional resistance is considerably reduced for workpieces that are subjected to sliding friction. The reduction of the so-called gas time to about half the hitherto usual time indicates that the duration of the etching attack of the phosphating solution, and thus the  
35 removal of metal from the workpiece, is considerably

reduced. It is assumed that the content of nitroguanidine in the phosphating solution leads to a certain passivation of the metal surface, which however permits a reduced etching attack and/or leads to an earlier start of the layer formation.

The invention is described in more detail with the aid of the following example.

Example:

Steel cup-shaped tappets were first of all degreased by immersion in a strongly alkaline, aqueous cleansing agent, next rinsed with water, then pre-rinsed in a slurry of finely divided manganese phosphate, and finally phosphated by immersion in a phosphating solution at 80°C for a duration of ten minutes.

The phosphating solution contained

20

11.8 g/l of manganese,  
0.5 g/l of nickel,  
1 g/l of iron(II),  
36 g/l of phosphate (calculated as  $P_2O_5$ )  
4.6 g/l of nitrate and  
0.36 g/l of citrate (calculated as citric acid).

25

The total point number of the phosphating solution was 80, and the point number of the free acid was 11 (measured with 60 g of concentrate per 1 l of water). For the determination of the total acid point number and free acid point number, reference should be made to the details given above.

30

Finely crystalline phosphate layers with a layer weight of 7 g/m<sup>2</sup>, corresponding to a layer thickness of 3 to 4 μm, and an averaged roughness depth R<sub>z</sub> of 1.3 to 2.4 μm, were  
10 obtained. The gas time was 2 to 3 minutes.

A comparative test was carried out under identical conditions with the above phosphating solution, which however did not contain nitroguanidine. Phosphate layers  
15 were formed which, although being finely crystalline, nevertheless had an averaged roughness depth  $R_z$  of 5 to 6  $\mu\text{m}$ . The layer weight was 6  $\text{g}/\text{m}^2$ . The gas time was 6 to 10 minutes.

## Patent Claims

1. Method for applying manganese phosphate layers to iron  
or steel surfaces using phosphating solutions  
5 containing manganese, phosphate or iron(II) ions as  
well as nitroguanidine, characterised in that in order  
to form a manganese phosphate layer having a minimum  
thickness of 2  $\mu\text{m}$  and an averaged maximum roughness  
depth ( $R_z$ ) of 2.5  $\mu\text{m}$  measured after drying, the  
10 workpieces are brought into contact with a phosphating  
solution containing  
  
0.2 to 4 g/l of iron(II) ions  
10 to 25 g/l of manganese ions  
15 25 to 50 g/l of phosphate ions (calc. as  $\text{P}_2\text{O}_5$ )  
3 to 35 g/l of nitrate ions  
0.5 to 5 g/l of nitroguanidine  
  
that has 7 to 24 points of free acid, 50 to 140 points  
20 of total acid, as well as an S value of 0.2 to 1.
2. Method according to claim 1, characterised in that the  
workpieces are brought into contact with a phosphating  
solution that contains 0.5 to 2 g/l of nitroguanidine.  
25
3. Method according to claim 1 or 2, characterised in  
that the workpieces are brought into contact with a  
phosphating solution, that contains at most 2.5 g/l of  
30 iron(II) ions.
4. Method according to claim 1, 2 or 3, characterised in  
that the workpieces are brought into contact with a  
phosphating solution, that in the case of the  
35 treatment of steel, contains a complex-forming agent

for the alloying constituents of the steel, preferably citric acid.

5. Method according to one or more of claims 1 to 4,  
5 characterised in that the workpieces are brought into contact with a phosphating solution that additionally contains
- 0.2 to 4 g/l of nickel ions
- or
- 10 0.2 to 4 g/l of magnesium ions.
6. Method according to one or more of claims 1 to 5,  
characterised in that the workpieces are brought into contact with a phosphating solution in which a  
15 proportion of the manganese ions are replaced by manganese carbonate in order to neutralise the free acid.
7. Use of the method according to one or more of claims 1  
20 to 6 for workpieces that are subjected to a sliding friction, such as axles, gear mechanism parts and engine pistons.



(12) NACH DEM VERTRAG ÜBER DIE INTERNATIONALE ZUSAMMENARBEIT AUF DEM GEBIET DES  
PATENTWESENS (PCT) VERÖFFENTLICHTE INTERNATIONALE ANMELDUNG(19) Weltorganisation für geistiges Eigentum  
Internationales Büro(43) Internationales Veröffentlichungsdatum  
5. April 2001 (05.04.2001)

PCT

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SEIFERT, Detlev [DE/DE]; Akazienweg 20, 64839 Münster (DE). STICKLER, Ralf [DE/DE]; Torgasse 11, 63796 Kahl am Main (DE).

(21) Internationales Aktenzeichen: PCT/EP00/09193

(22) Internationales Anmeldedatum:  
20. September 2000 (20.09.2000)

(74) Anwälte: UPPENA, Franz usw.; Dynamit Nobel Aktiengesellschaft, Patentabteilung, 53839 Troisdorf (DE).

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(84) Bestimmungsstaaten (regional): europäisches Patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).

(30) Angaben zur Priorität:  
199 47 232.7 30. September 1999 (30.09.1999) DE

Veröffentlicht:

— Mit internationalem Recherchenbericht.

— Vor Ablauf der für Änderungen der Ansprüche geltenden Frist; Veröffentlichung wird wiederholt, falls Änderungen eintreffen.

(71) Anmelder (für alle Bestimmungsstaaten mit Ausnahme von US): CHEMETALL GMBH [DE/DE]; Trakehner Strasse 3, 60487 Frankfurt am Main (DE).

Zur Erklärung der Zweibuchstaben-Codes, und der anderen Abkürzungen wird auf die Erklärungen ("Guidance Notes on Codes and Abbreviations") am Anfang jeder regulären Ausgabe der PCT-Gazette verwiesen.

(72) Erfinder; und

(75) Erfinder/Anmelder (nur für US): NITTEL, Klaus-Dieter [DE/DE]; Raabstrasse 8, 60431 Frankfurt am Main (DE).

(54) Title: METHOD FOR APPLYING MANGANESE PHOSPHATE LAYERS

(54) Bezeichnung: VERFAHREN ZUM AUFBRINGEN VON MANGANPHOSPHATSCHICHTEN

(57) Abstract: The invention relates to a method for applying manganese phosphate layers on iron or steel surfaces. The aim of the invention is to produce a manganese phosphate layer with a minimum thickness of 2 µm and an average surface roughness (R<sub>a</sub>) of 2.5 µm measured after drying. To this end, a phosphatizing solution is used that contains 0.2 to 4 g/l ferrous ions, 10 to 25 g/l manganese ions, 25 to 50 g/l phosphate ions (calculated as P<sub>2</sub>O<sub>5</sub>), 3 to 35 g/l nitrate ions, 0.5 to 5 g/l nitroguanidine, and that has 7 to 24 free acid points, 50 to 140 total acid points and an S value of 0.2 to 1. The phosphatization solution preferably contains 0.5 to 2 g/l nitroguanidine and maximally 2.5 g/l ferrous ions and additionally 0.2 to 4 g/l nickel ions or 0.2 to 4 g/l magnesium ions. The inventive method is especially applied to work pieces that are subject to a sliding friction.

(57) Zusammenfassung: Bei einem Verfahren zum Aufbringen von Manganphosphatschichten auf Eisen- oder Stahloberflächen bringt man zwecks Ausbildung einer Manganphosphatschicht mit einer Mindestdicke von 2 µm und einer gemittelten maximalen Rauhtiefe (R<sub>a</sub>) von 2,5 µm - gemessen nach dem Trocknen - Werkstücke mit einer Phosphatierungslösung, die 0,2 bis 4 g/l Eisen (II) - Ionen, 10 bis 25 g/l Manganionen, 25 bis 50 g/l Phosphationen (ber. als P<sub>2</sub>O<sub>5</sub>), 3 bis 35 g/l Nitrationen, 0,5 bis 5 g/l Nitroguanidin enthält, 7 bis 24 Punkte Freie Säure, 50 bis 140 Punkte Gesamtsäure sowie einen S-Wert von 0,2 bis 1 aufweist. Vorzugsweise enthält die Phosphatierungslösung 0,5 bis 2 g/l Nitroguanidin und maximal 2,5 g/l Eisen (II) - Ionen sowie zusätzlich 0,2 bis 4 g/l Nickelionen oder 0,2 bis 4 g/l Magnesiumionen. Das Verfahren findet insbesondere Anwendung auf Werkstücke, die einer gleitenden Reibung ausgesetzt sind.

WO 01/23638 A1

7

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

## METHOD FOR APPLYING MANGANESE PHOSPHATE LAYERS

the specification of which:

( ) is attached hereto.

(X) was filed on March 21, 2002 as U.S. Serial No 10/088,840.

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, Section 119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

<u>199 47 232.7</u>	<u>Germany</u>	<u>30 September 1999</u>	<u>Yes (X)</u>	<u>No ( )</u>
(Number)	(Country)	(Day/Month/Year Filed)	Priority Claimed	

### U.S. Priority Applications

U.S. Priority Applications  
I hereby claim the benefit under Title 35, United States Code, Section 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of the application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, Section 112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, Section §1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

<b>PCT/EP00/09193</b>	<b>September 20, 2000</b>	<b>Pending</b>
(Applic. Serial No.)	(Filing Date)	(Status patented/pending/abandoned)
<b>(Applic. Serial No.)</b>	<b>(Filing Date)</b>	<b>(Status patented/pending/abandoned)</b>

CHEMNT-206

POWER OF ATTORNEY

7 I hereby appoint the following attorneys to prosecute this application and transact all business in the Patent and Trademark Office connected therewith: Peter F. Felfe, Reg. No. 20,297; Norman D. Hanson, Reg. No. 30,946; John A. Bauer, Reg. No. 32,554; No. 36,669; James R. Crawford, Reg. No. 39,155; Andrew Im, Reg. No. 40,657, and David Rubin, Reg. No. 40,314, my attorneys with full power of substitution and revocation. Address all telephone calls to James R. Crawford, (212) 318-3148. Address all correspondence to James R. Crawford, Esq. at

**FULBRIGHT & JAWORSKI L.L.P.**

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New York, NY 10103

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

1-00 (1) Klaus-Dieter NITTEL Klaus-Dieter Nitte 04/04/02  
Full Name/Sole or First Inventor Signature Date

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